

PHYSICAL-CHEMICAL PROPERTIES OF SPINNING SOLUTIONS FOR ALUMINUM-SILICATE FIBROUS MATERIALS

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The rheological properties and aggregative stability of spinning solutions for obtaining aluminum-silicate fibrous materials are examined. It is shown that the acetate groups in polyvinyl alcohol affect the properties of the spinning solutions.

Key words: aluminum-silicate fibrous materials, spinning solution, polyvinyl alcohol, acetate groups, aggregative stability, rheological properties.

The process of obtaining polycrystalline aluminum-silicate fibrous materials by solution technology includes obtaining homogeneous solutions which are subsequently transformed into a solid uniform xerogel [1, 2]. The solutions must not contain any nonuniformity that softens the materials. In the process of concentration and transformation of the solutions into xerogel moisture is removed and the concentration of the main components increases; this can result in mutual destabilization of the solutions. In this connection, the spinning solutions (SS) must not lose aggregative stability and must not contain gel nonuniformities during preparation and concentration [3]. One of the main functions of polyvinyl alcohol (PVA) is to stabilize the spinning composition. In the present work the stabilizing power and surface activity of PVA and its capacity to form the required rheological properties of SS depending on the mass fraction of acetate groups present in them is examined.

Samples of PVA with content of residual acetate groups from 7 to 18 wt.% and close values of the dynamic viscosity 4% of the solution (molecular mass) were used to investigate the effect of polyvinyl alcohol on the properties of SS.

The samples obtained were used to prepare spinning solutions for aluminum-silicate fibrous materials. The compatibility of the components was observed visually. The aggregative stability of the spinning solutions was investigated with a KFK-3 photometer at wavelength 670 nm. The change in the optical density in time served as the criterion for the aggregative stability of the solutions. The rheological properties of the spinning solutions were determined with a MCR-501 rheometer in the rotation and oscillation regimes [4–6]. The surface tension was determined by the method of

ring detachment in a KRÜSS K-20 tensiometer at temperature $25 \pm 0.5^\circ\text{C}$.

The aggregative stability time of the samples of the PVA-based spinning solutions with acetate group content < 10 wt.% at 25°C is 11 days, and for acetate group content 15% the stability time of the solutions is at least 16 days (Fig. 1). An increase in the temperature of the SS results in sharp destabilization of the spinning composition (Fig. 2). At 40°C the stability time of the spinning solutions is 38 h (acetate group content 8 wt.%) and 72 h (acetate group content 15%). At 80°C SS with PVA and acetate group content $< 10\%$ forms a solid phase in the process of being concentrated, while SS with PVA and acetate group mass fraction from 10 to 15% remain stable for 48 h, which is adequate for concentrating the solutions and drying the materials. The increase in the aggregative stability time of solutions with the content of the acetate groups in PVA increasing from 10 to 15% is explained by a decrease in the fraction of OH-groups

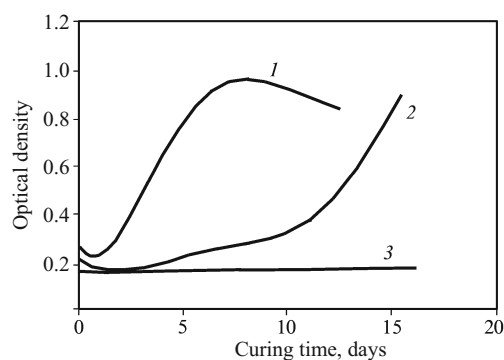


Fig. 1. Optical density of spinning solutions ($\lambda = 670$ nm) versus the curing time at temperature 25°C . Mass fraction of acetate groups in PVA: 1) 8%; 2) 11%; 3) 15%.

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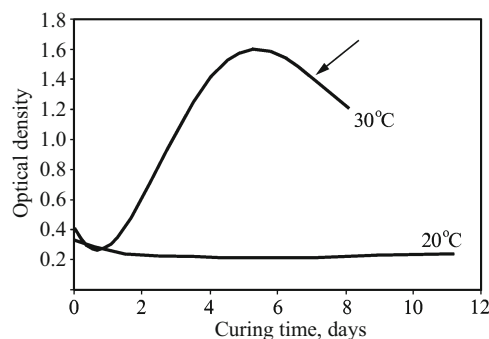


Fig. 2. Time variation of the optical density of spinning solutions for different values of the temperature. The arrow marks the precipitation time.

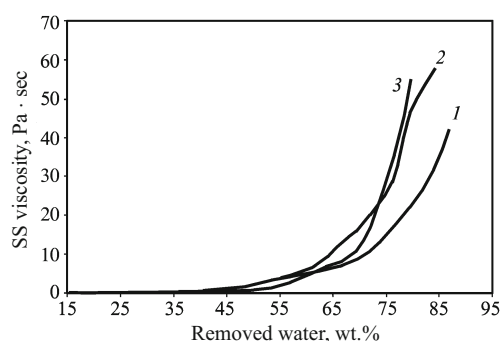


Fig. 3. Viscosity versus the amount of evaporated water for PVA samples with different acetate group mass fraction: 1) 8%; 2) 11%; 3) 15%.

capable of entering into interaction with the components of the SS as well as by the spatial factor which impedes the chemical interaction of the components. As the content of acetate groups continues to increase the aggregative stability decreases because the solubility of polyvinyl alcohol decreases.

An investigation of the effect of the acetate group content on the concentration process for spinning solutions showed that it increases fraction in PVA increases in the process of concentration the viscosity increases more rapidly and a smaller amount of water must be removed from the solution in order to achieve a prescribed viscosity (Fig. 3). This is favorable to the aggregative stability of SS and the efficiency of the concentration process.

The rheological properties of spinning solutions also depend on the acetate group content. As the acetate group content in PVA increases, the stability of the dynamic viscosity of the solutions increases in time (Fig. 4a) and the activation energy of viscous flow and hysteresis of the stresses of the spinning solutions decrease (Fig. 4b and c). This is associated with a decrease of the chemical interaction of PVA with the components of the SS, which results in the formation of three-dimensional branching linkages.

A decrease in the hysteresis area indicates that a stronger structure of the spinning solution forms [6]. This is con-

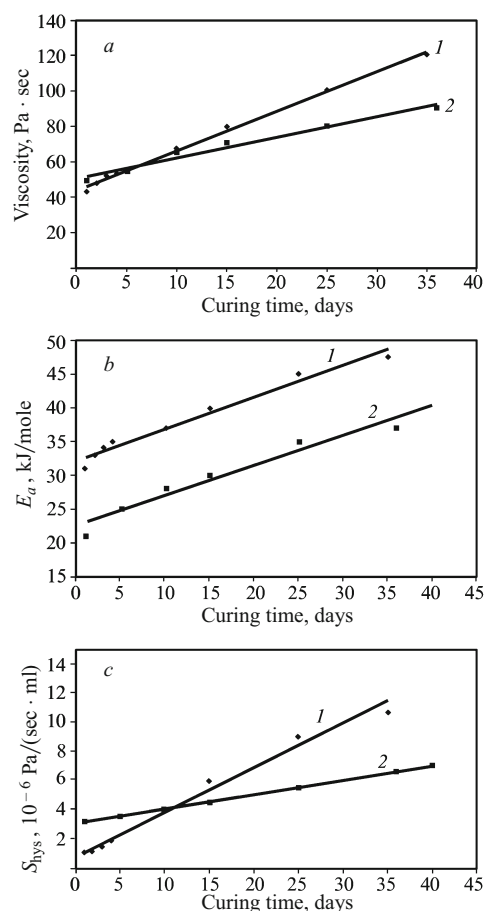


Fig. 4. The rheological properties of spinning solutions during the curing process. Mass fraction of acetate groups in PVA: 1) 15%; 2) 18%; a) change in viscosity; b) change in the activation energy E_a ; c) change in the hysteresis area S_{hys} .

firmed by tests for breakdown — restoration of the structure of the solutions. The degree of breakdown of the viscous and elastic components equals 1 and 9%, respectively, for SS containing PVA with acetate group mass fraction 18% while the degree of breakdown of the viscous and elastic components of the SS containing PVA with acetate group mass fraction 15% equals 6 and 22%, respectively, i.e., an increase of the acetate group content in PVA by 3% decreases the degree of breakdown of the viscous component of the SS six-fold and the elastic component 2.5-fold.

It was established that the surface tension of the spinning solutions using all PVA samples has approximately the same values in the interval 40 – 46 mN/m. However, it was noted that the surface tension tends to decrease with increasing mass fraction of the acetate groups (Fig. 5). That is, the change in the acetate group content in PVA does not have a significant effect on the surface activity of the latter, but results in some change in the molecular bonds in the spinning solution.

It can be concluded from the results of these investigations that for the formation of fibrous materials it is best to

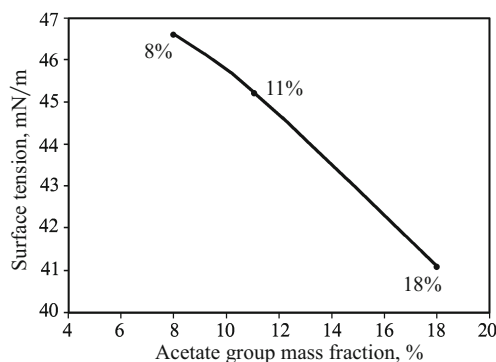


Fig. 5. Surface tension of spinning solutions versus the mass fraction of acetate groups in PVA.

use PVA with the maximum content of acetate groups, which is limited by the solubility of PVA in water or other appropriate solvent.

It can be concluded that for the formation of fibrous materials it is best to use PVA with the maximum content of acetate groups, which is limited by the solubility of PVA in water or other appropriate solvent.

CONCLUSIONS

The relationship between the content of residual acetate groups and the physical-chemical properties of spinning so-

lutions was determined for aluminum-silicate fibrous materials.

It was shown that to obtain the best aggregative stability and the minimum surface tension of spinning solutions the mass fraction of acetate groups in PVA must be in the range 11 – 18%.

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